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METHOD FOR DETERMINATION OF GOLD AND SILVER IN GOLD AND SILVER THREAD AND EMBROIDERY MATERIALS

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Indian Standard

METHOD FOR DETERMINATION OF GOLD AND SILVER IN GOLD AND SILVER THREAD AND EMBROIDERY MATERIALS

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Indian Standard

METHOD FOR DETERMINATION OF GOLD AND SILVER IN GOLD AND SILVER THREAD AND EMBROIDERY MATERIALS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 31 August 1981, after the draft finalized by the Precious Metals Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 A number of standards have been published on gold and silver thread and embriodery materials. Need was, therefore, felt to prescribe suitable methods for estimation of gold and silver in such materials.

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard covers the method for estimation of gold and silver in gold and silver thread and embroidery materials.

2. QUALITY OF REAGENTS

2.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which effect the results of analysis.

3. ESTIMATION OF GOLD

3.1 Outline of the Method — Gold is weighed in metallic state and determined in the form of mass obtained by the nitric acid treatment of the sample.

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*Rules for rounding off numerical values (*revised*).

†Specification for water for general laboratory use (*second revision*).

3.2 Reagents

3.2.1 *Dilute Nitric Acid* — rd = 1·2 and 1·20 (*v/v*).

3.2.2 *Concentrated Hydrochloric Acid* — rd = 1·16 (conforming to IS : 265-1976*).

3.2.3 *Concentrated Nitric Acid* — rd = 1·42 (conforming to IS : 264-1976†).

3.2.4 *Aqua Regia* — Mix concentrated hydrochloric acid and concentrated nitric acid in the ratio of 3 : 1.

3.2.5 *Concentrated Ammonium Hydroxide* — 20 percent.

3.2.6 *Sulphurous Acid* — Make approximately 5 percent solution of sulphur dioxide in water.

3.3 Procedure‡

3.3.1 Transfer about 5 g of an accurately weighed sample to a porcelain crucible and ignite to burn away completely the organic matter present. Digest with 20 ml of dilute nitric acid (rd 1·2) in a beaker. Boil off the brown fumes and dilute the solution to 100 ml with water. Warm the solution, filter through Whatman filter paper No. 30 and wash the residue with dilute nitric acid (1 : 20) and finally with water until the washings are free from acid. Preserve the filtrate for the estimation of silver.

3.3.2 Dissolve the residue in 10 ml of aqua regia and evaporate to dryness on a water-bath. Repeat evaporation to dryness twice more with a few millilitres of concentrated hydrochloric acid to remove nitric acid completely. Take up with 50 ml of water and filter, if necessary. Wash thoroughly with water. Preserve the filtrate.

3.3.3 Add 5 ml of ammonium hydroxide over the residue on the filter paper, if any. Wash with hot water three times. Preserve the filtrate for estimation of silver and discard the residue, if any.

3.3.4 Add 30 ml of sulphurous acid to the solution reserved for estimation of gold under **3.3.2**. Stir well and keep overnight. Filter the precipitate through Whatman filter paper No. 30 and wash with cold

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*Specification for hydrochloric acid (*second revision*).

†Specification for nitric acid (*second revision*).

‡In case of embroidery materials, step to ignite the sample to burn away the organic matter should be omitted. The sample after cleaning with alcohol is weighed accurately and then digested with nitric acid as in **3.3.1**. Further complete the estimation as in **3.3.2** to **3.4**.

water. Dry the residue and transfer it to a tared porcelain crucible. Ignite in a muffle furnace at 900°C to constant mass.

3.4 Calculation — Calculate the gold content as follows:

$$\text{Gold, percent} = \frac{A}{B} \times 100$$

where

A = mass in g of the ignited residue, and
B = mass in g of the sample taken.

4. ESTIMATION OF SILVER

4.1 By Silver Chloride Precipitation Method

NOTE — Every precaution shall be taken to avoid contamination by chlorides from any source.

4.1.1 *Outline of the Method* — This method is based on the precipitation of silver as silver chloride in dilute nitric acid solution.

4.2 Reagents

4.2.1 *Dilute Hydrochloric Acid* — 3·97 (*v*/*v*).

4.2.2 *Dilute Nitric Acid Wash Solution* — 1 : 200 (*v*/*v*).

4.2.3 *Ethyl Alcohol* — 95 percent (*v*/*v*).

4.2.4 *Dilute Nitric Acid* — rd = 1·2.

4.3 Procedure

4.3.1 *For Gold Thread* — Mix both the filtrates reserved under **3.3.1** and **3.3.3** and dilute to 175 ml with water, heat to boiling, and add dilute hydrochloric acid dropwise from a pipette until silver chloride precipitates out completely. Wrap the beaker with black paper to prevent exposure to light. Let the solution cool, and the precipitate settle. Add few drops of dilute hydrochloric acid to the supernatant liquid to ascertain whether the precipitation is complete.

4.3.2 Filter on the weighed sintered glass crucible, washing the precipitate several times with dilute nitric acid wash solution and then wash once with cold water. Finally wash the precipitate with alochol and dry at 110 to 130°C to constant mass.

4.3.3 *For Silver Thread* — In case of silver thread, take 5 g of the sample in a porcelain crucible and ignite to burn away the organic matter completely. Record the weight. Digest with 20 ml of dilute nitric acid, boil off the brown fumes and dilute the solution to 175 ml with water. Filter through paper No. 30 and carry out the procedure as in **4.3.1** and **4.3.2.**

4.4 Calculation — Calculate the percentage of silver as follows:

$$\text{Siver, percent} = A \times \frac{0{\cdot}752\,6}{B} \times 100$$

where

A = mass in g of silver chloride, and
B = mass in g of the sample taken.

4.5 By Volhard's Potassium Thiocyanate (Alternate) Method

4.5.1 *Outline of the Method* — The method is based on the difference between the solubilities of thiocyanates of iron and silver. When all the silver has been precipitated as the white insoluble thiocyanate, the ferric indicator reacts with the thiocyanate producing the characteristic red colour.

4.6 Reagents

4.6.1 *Dilute Nitric Acid* — 1 : 1 (*v/v*).

4.6.2 *Ferric Ammonium Sulphate Indicator Solution* — Prepare 100 ml of a saturated solution of this reagent in water and add sufficient colourless nitric acid to clear up the solution, producing pale yellow colour.

4.6.3 *Standard Silver Solution* (1 ml = 0·005 g *of Ag*) — Dissolve one g of pure silver foil in 10 ml of dilute nitric acid (rd = 1·2). Boil to expel the brown fumes and dilute to 200 ml.

4.6.4 *Standard Potassium Thiocyanate Solution* — 0·1 *N*. Dissolve 4·6 g of potassium thiocyanate in water and dilute to 500 ml. Standardize against standard silver solution (*see* **4.6.3**) by following the procedure given in **4.7.1**.

4.7 Procedure

4.7.1 Transfer the filtrate reserved under **3.3.1** and **3.3.3** to a 250-ml volumetric flask and make up the solution to the mark by diluting with water. Pipette out 50 ml of the solution to a conical flask, add 10 ml of dilute nitric acid and 2 to 3 ml of the indicator solution and titrate with standard potassium thiocyanate solution until a permanent red tint is obtained.

4.8 Calculation — Calculate the percentage of silver as follows:

$$\text{Silver, percent} = \frac{A \times B \times 0{\cdot}107\,88 \times 5}{C} \times 100$$

where

A = volume in ml of the standard potassium thiocyanate solution required,

B = normality of the standard potassium thiocyanate solution, and

C = mass in g of the sample taken.

INDIAN STANDARDS ON PRECIOUS METALS

IS:	
639-1965	Gold leaf (*revised*)
1417-1981	Grades of gold and gold alloys (*second revision*)
1418-1972	Method for assaying of gold in gold and gold alloys (*first revision*)
1953-1973	Chemical Analysis of silver anodes (*first revision*)
2112-1981	Grades of silver and silver alloys (*first revision*)
2113-1972	Methods for assaying of silver in silver and silver alloys (*first revision*)
2270-1965	Methods for assaying of platinum and platinum alloys
2275-1963	Grades of platinum
2279-1963	Fine silver bar, sheet, wire, granules and token (*lagdi or mohur*)
2767-1964	Gold thread (*silver base*)
2790-1981	Guidelines for manufacture of 23·3, 22, 21, 18, 14, 12 and 9 carat gold (*first revision*)
3088-1967	Method for assaying of fine grade palladium
3095-1981	Guidelines for the manufacture of Solders for use in goldware (*first revision*)
3096-1965	Fine grade palladium
3110-1965	Silver leaf
3111-1965	Silver thread
3112-1965	Gold and silver embroidery materials
3541-1981	Code of practice for manufacture of 23 and lower carat gold alloys (*first revision*)
3571-1966	Dental gold solders
3578-1966	Dental gold alloys wire
3610-1966	Dental gold foil
4134-1967	Reeommended colour classification of rough diamond
4704-1968	Silver-tin dental amalgam alloy
4705-1968	Dental mercury
4799-1969	Dental casting gold alloys
5320-1969	Fine silver ingot
5954-1970	Dental white gold alloys
6018-1971	Platinum, Platinum-rhodium catalyst gauzes
6019-1971	Platinum disk
6070-1971	Platinum crucible and lid
6683-1973	Diameters for wires of platinum group metals and their alloys
6882-1973	Plntinum electrodes
6889-1973	Chemical analysis of silver-tin dental alloys
6890 (Part I)-1973	Chemical analysis of dental gold alloys: Part I Determination of gold, silver, platinum, palladium and copper
6890 (Part II)-1975	Chemical analysis of Dental gold alloys: Part II Determination of nickel and zinc
7225-1974	Dental cobalt chromium casting alloys
7255 (Part I)-1974	Methods of chemical analysis of solders for use in gold ware: Part I Determination of gold, silver and copper
7255 (Part II)-1977	Chemical analysis of solders for use in gold ware: Part II Determination of cadmium and zinc by polarographic method
7562-1974	Gold cladding
8866-1978	Guidelines for marking purity of gold on gold articles/ornaments
8929-1978	High purity gold wire for electrical contacts.